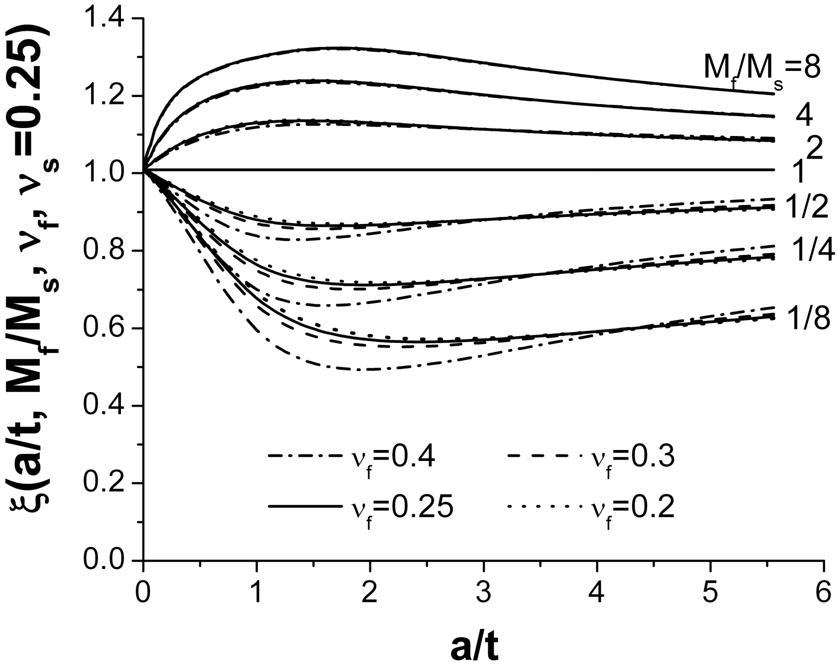
Determining the Elastic Modulus and Hardness of an Ultrathin Film on a Substrate Using Nanoindentation

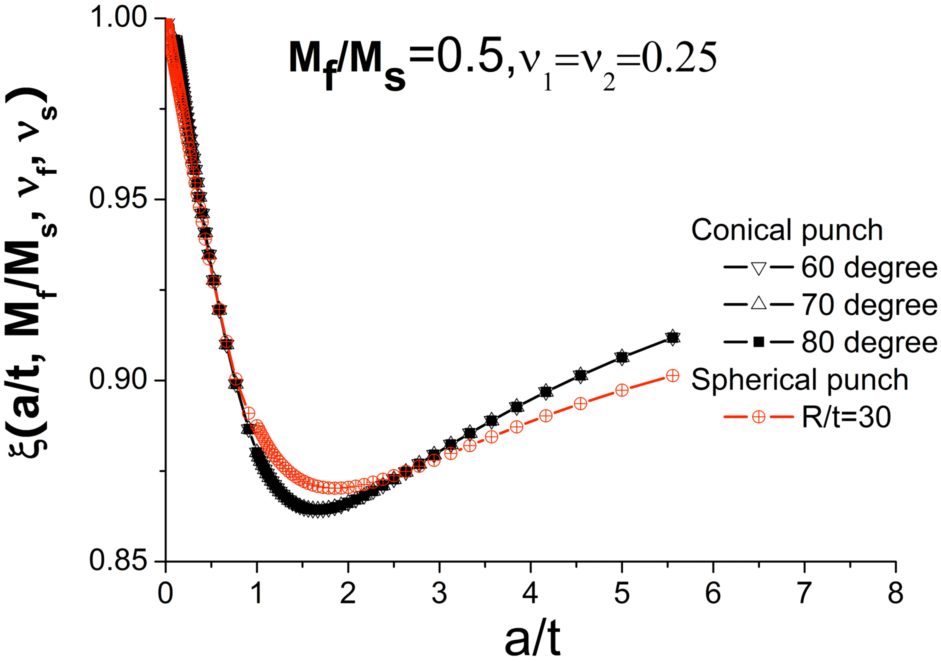
Citation
Li, Han, and Joost J. Vlassak. 2009. Determining the elastic modulus and hardness of an ultrathin film on a substrate using nanoindentation. Journal of Materials Research 24(3): 1114-1126.

Published Version
doi:10.1557/JMR.2009.0144

Figures



(a)



(b)

Fig. 1. The dimensionless correction factor ξ for an elastic indentation as a function of normalized contact radius for (a) different elastic mismatch and a conical indenter, and for (b) various indenter shapes.

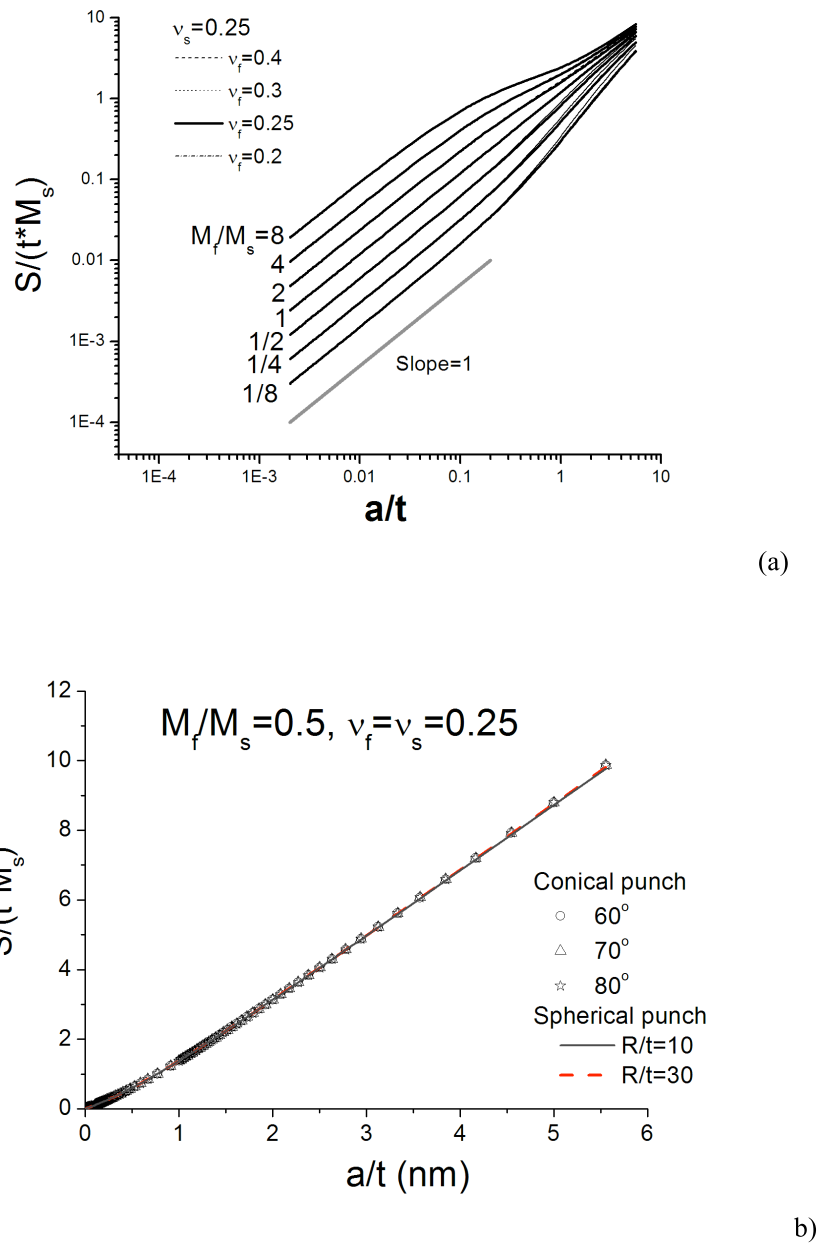


Fig. 2. Normalized contact stiffness versus contact radius calculated from Yu's solution for (a) different elastic mismatches, and (b) various conical and spherical punches.

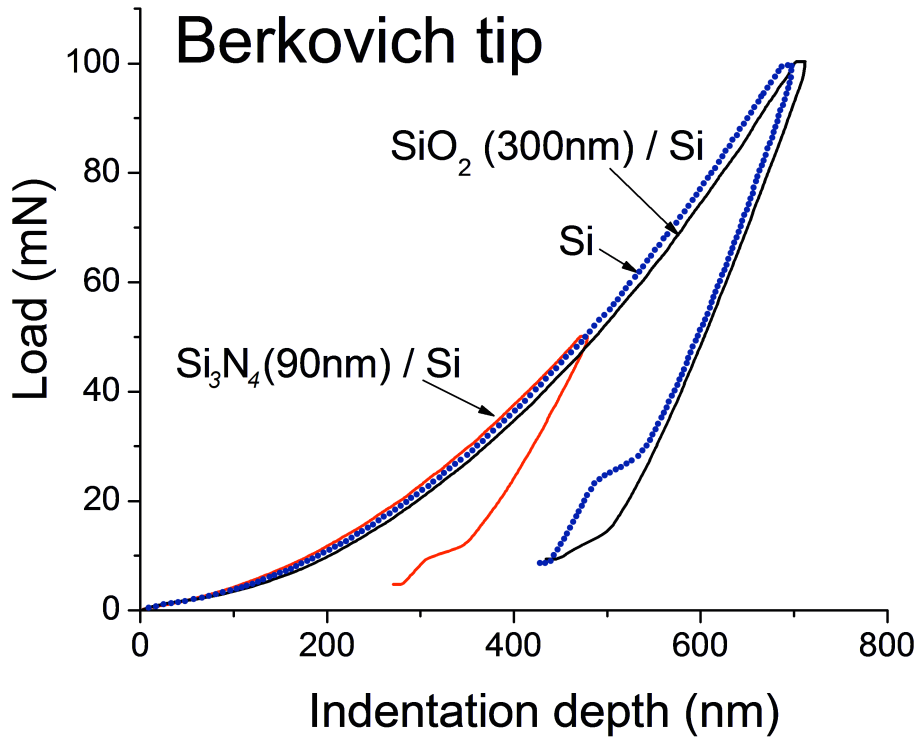


Fig. 3. Experimental load-displacement curves for the Si_3N_4 and SiO_2 films, and for the silicon substrate.

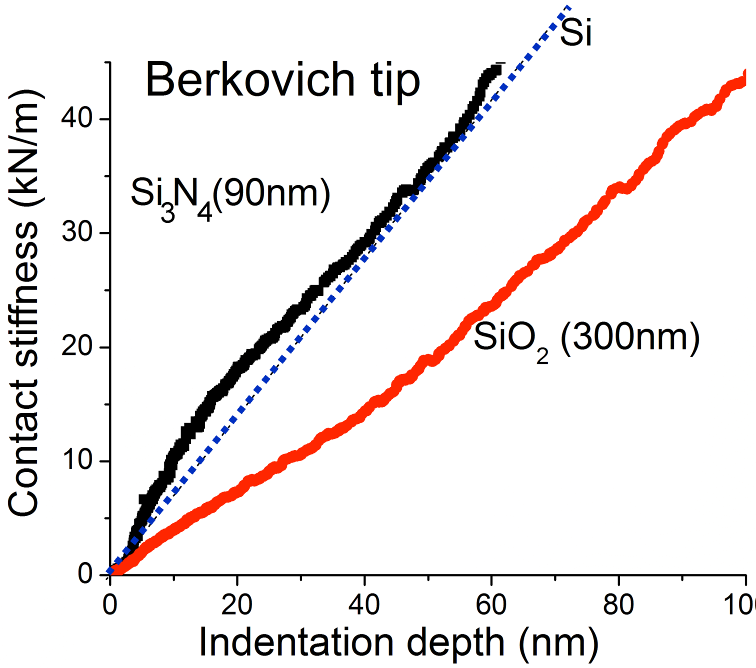


Fig. 4. Curves of the experimental contact stiffness versus indentation depth for the Si_3N_4 film, the SiO_2 film, and for the silicon substrate.

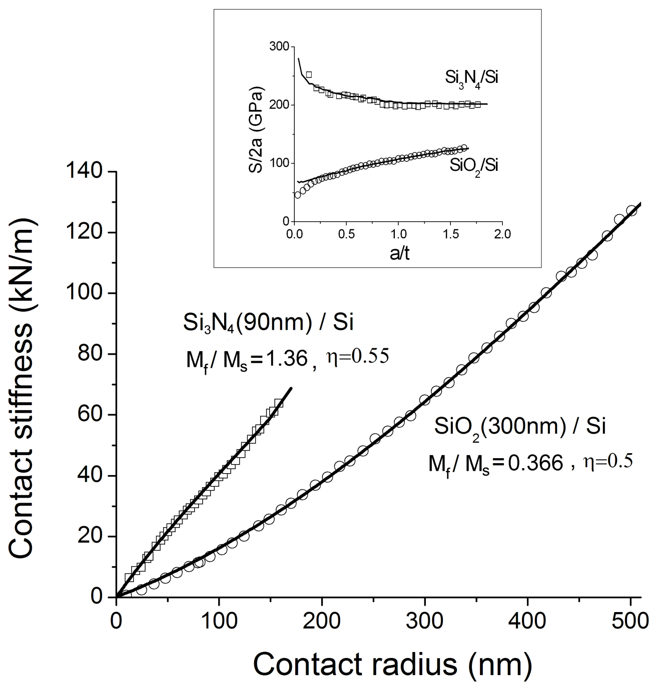


Fig. 5. Experimental (markers) and theoretical (solid curves) contact stiffness versus contact radius for the Si_3N_4 and SiO_2 samples. The inset presents the same data in the form of *S/2a* versus *a/t*.

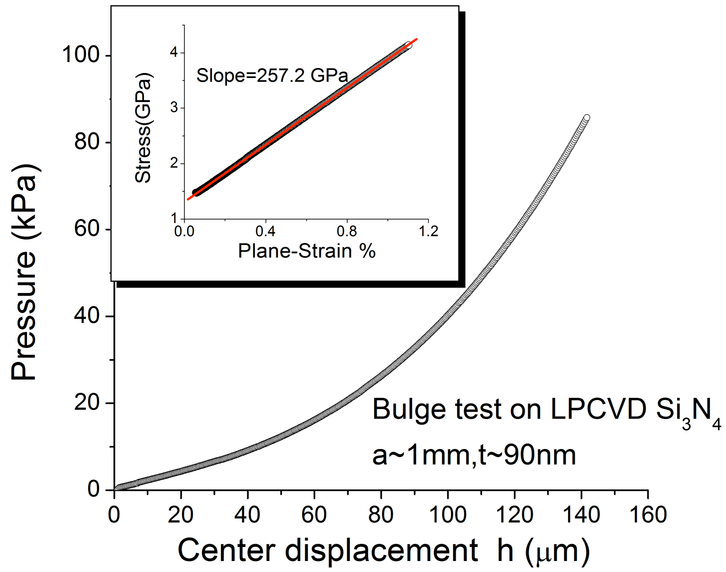


Fig. 6. The pressure-displacement curve for a freestanding LPCVD silicon nitride film obtained in the bulge test. The inset is the corresponding plane-strain stress-strain curve, yielding a plane-strain modulus of 257.2±1.5 GPa.

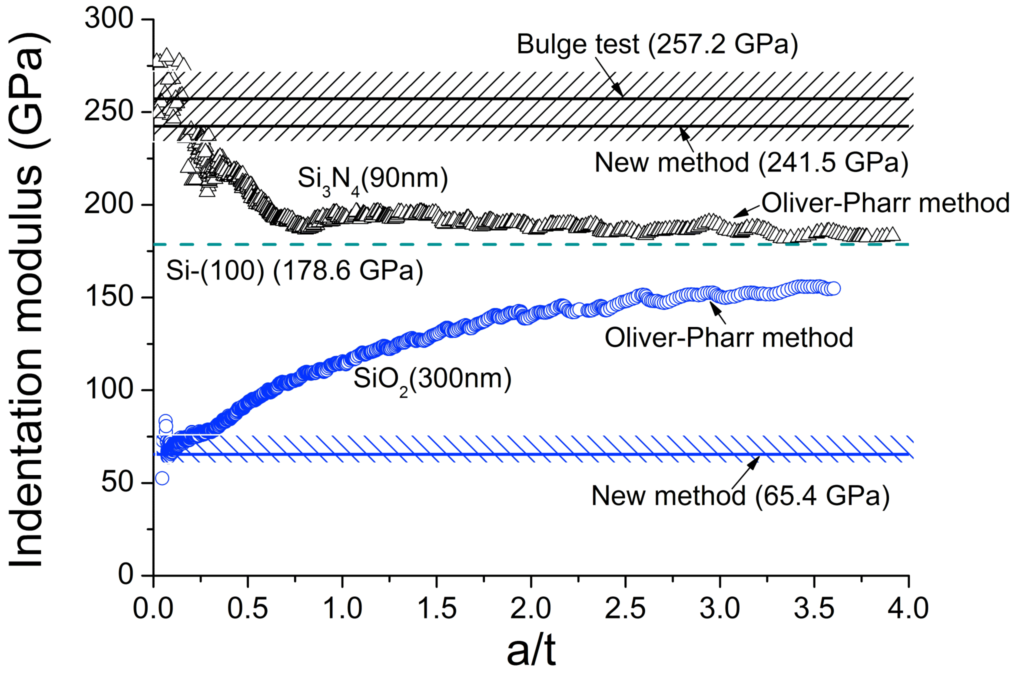


Fig. 7. The indentation modulus obtained with the Oliver-Pharr method as a function of contact radius normalized by film thickness, compared with the results obtained using the new method. The shaded regions represent the ranges of the SiO_2 and Si_3N_4 indentation moduli reported in the literature.

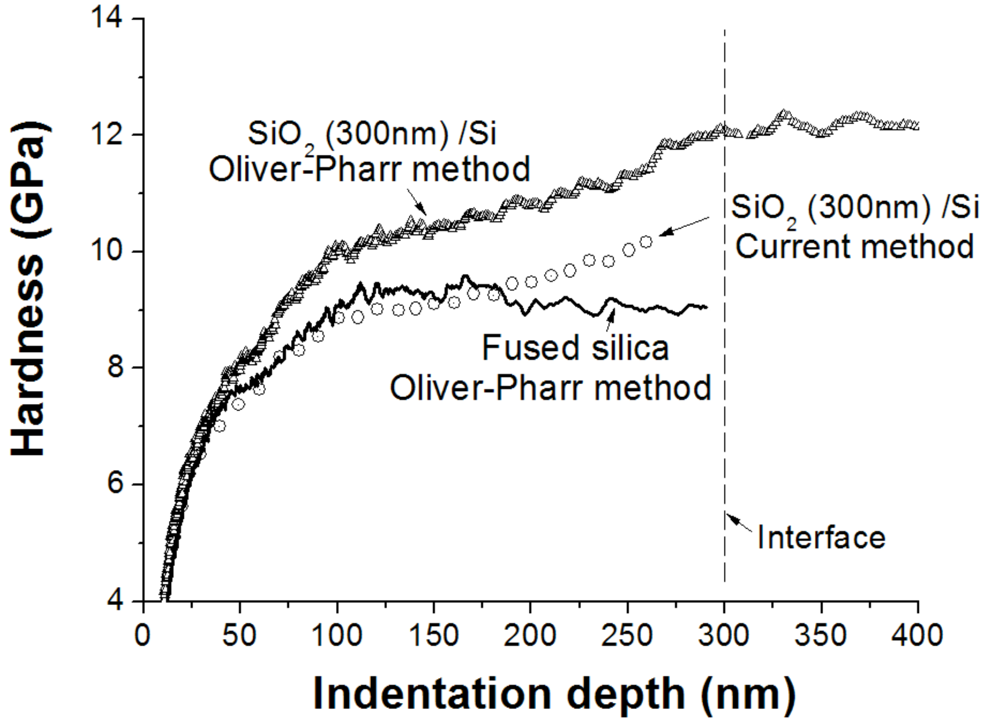


Fig. 8. The hardness of the SiO_2 film as a function of indentation depth calculated using several methods. The hardness of bulk fused quartz is included for comparison.

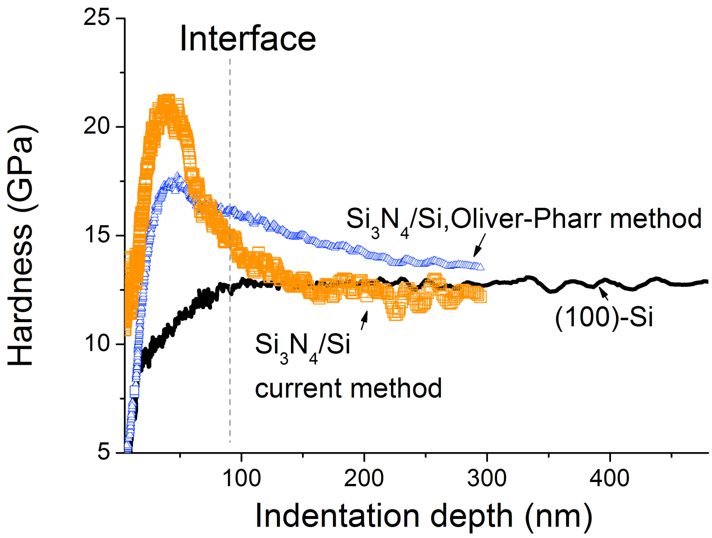


Fig. 9. The hardness of the Si_3N_4 film as a function of indentation depth calculated using several methods. The hardness for the silicon substrate is included for comparison.

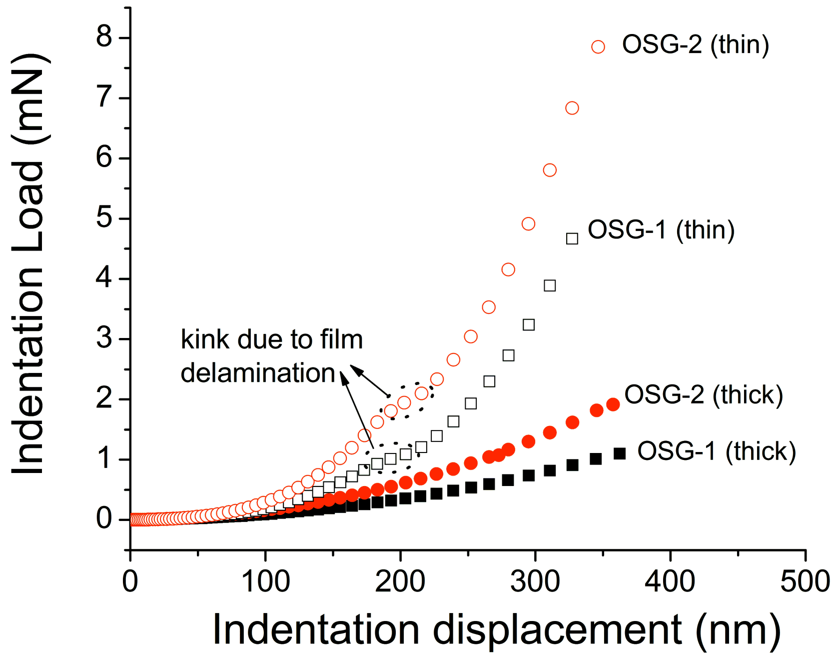


Fig. 10. Load-displacement curves for the two OSG films of the same properties but different thicknesses on silicon substrate, interfacial delamination at position circled.

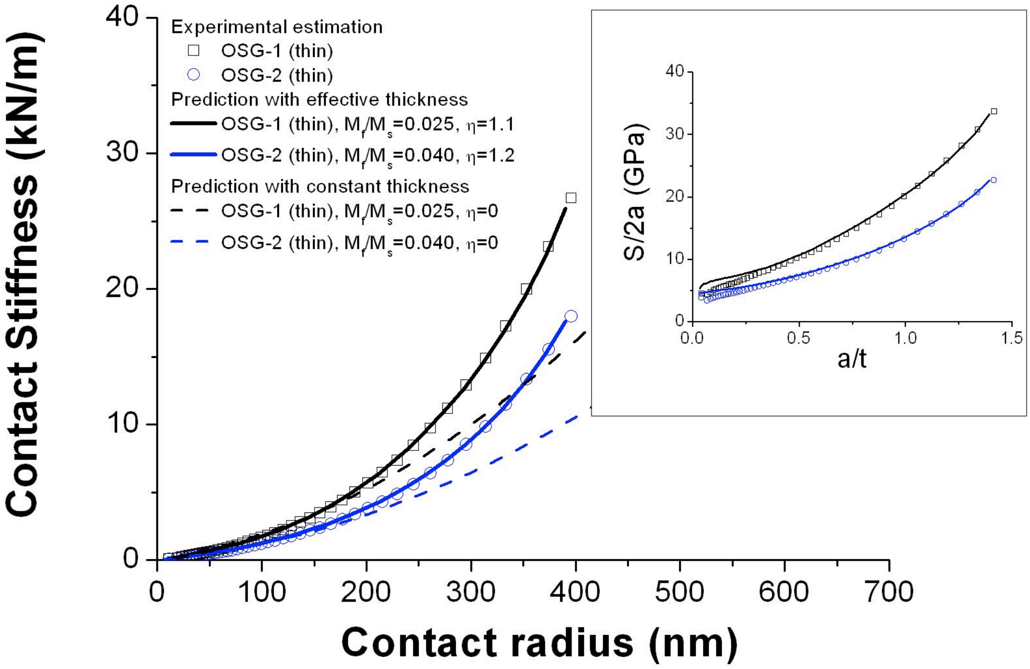


Fig. 11. Experimental and theoretical contact stiffness as a function of contact radius for the various OSG films. The inset presents the same data in the form of *S/2a* versus *a/t*.

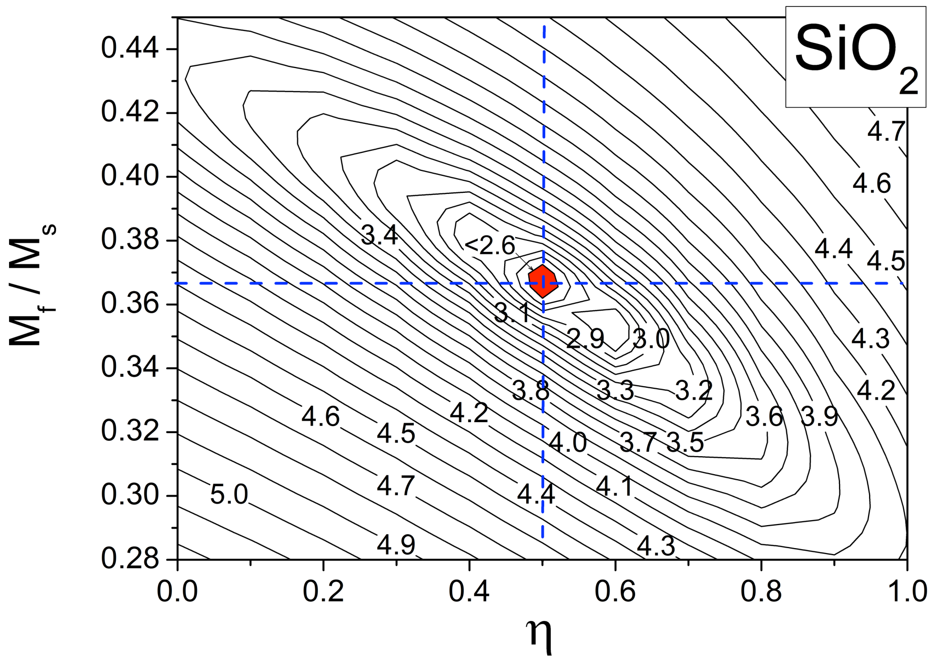


Fig. 12. Contour plot of $\log_{10}(\chi^2)$ as a function of M_f/M_s and η for the SiO_2/Si sample, with minimum falling within the highlighted region. The unit of χ^2 is in nm^2.

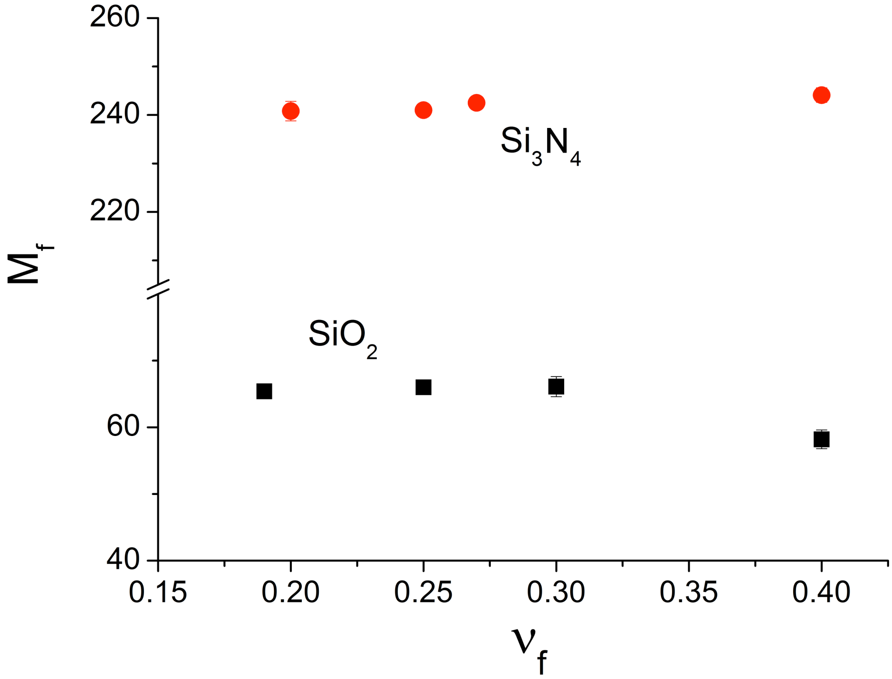


Fig. 13. Indentation moduli of the SiO_2 and Si_3N_4 films as a function of the value of Poisson's ratio assumed in the data analysis.

Tables

	Materials	Thickness (nm)
Compliant film	Thermally grown SiO_2	300 ± 10
Stiff film	LPCVD Si_3N_4	90 ± 2
Porous OSG films	OSG-1	280 ± 5
		2300 ± 20
	OSG-2	280 ± 5
		2300 ± 20

Table 1. Summary of materials for films and substrate investigated.

Materials	**Poisson's ratio**	***M (GPa)***	η	R^2
(100)-Si	*0.22* [12]	178.6±1.7	-	-
SiO_2 film	*0.19* [22~26]	65.4±0.7	0.50	0.9996
Si_3N_4 film	*0.27* [21]	242.5±0.9	0.55	0.9998
OSG-1 (thin)	*0.25*	4.45±0.19	1.10	0.9992
OSG-1 (thick)	0.25	4.50±0.20	-	-
OSG-2 (thin)	*0.25*	7.07±0.46	1.20	0.9995
OSG-2 (thick)	*0.25*	7.35±0.42	-	-

Table 2. Summary of the nanoindentation results for the various thin-film systems.

Film processing	Thickness (nm)	Method	Young's modulus (GPa)
Thermally grown below 1000°C (present work)	300±10	Nanoindentation	63.1±0.7
Thermally grown at 875~1200°C [22]	200~2000	Bulge test	65.2*
Thermally grown at 960°C [23]	80	Micro-beam resonance	67
Thermally grown [24]	325	Electrically activated membrane	69±14
Thermally grown at 1200°C [25]	650	Cantilever beam technique using X-ray diffraction	51.3*
Thermally grown at 1000°C [26]	1000	Brillouin light scattering technique	72
Bulk fused silica [1]	--	Nanoindentation	69.3

* Assume $\nu = 0.19$

Table 3. A survey of Young's moduli for thermally grown SiO_2 films reported in the literature.

Appendix: Step-by-step instructions to implement the proposed method

I. Steps to get the experimental $a \sim S$ relation as function of M_f and η

1. Assume initial values of M_f and η
2. Calculate the effective thickness t_{eff} for a given point on the indentation loading curve using Eq. 16.
3. Obtain the experimental value of the contact radius at this loading point by solving the following implicit equation numerically:

$$a_{\exp} = \sqrt{\frac{1}{\pi} f\left[h - \xi\left(\left.\frac{a_{\exp}}{t_{eff}}\right|_h, \frac{M_f}{M_s}\right)\varepsilon\frac{P}{S}\right]}, \tag{A1}$$

   where f is the area function of the indenter tip and ξ is obtained from Yu's solution.

4. Calculate the reduced stiffness to remove the compliance of the indenter tip using

$$S_r = \left(\frac{1}{S} - \frac{1}{S_{tip}}\right)^{-1}, \tag{A2}$$

   where $S_{tip} = 2a_{\exp}M_{tip}$ and M_{tip} =1146.6 GPa for a diamond indenter.

5. Repeat steps 2 through 4 for every point of the indentation loading curve to obtain the experimental $\left[S_r, a_{\exp}\right]$ relation for the values of M_f and η assumed in step 1.

II. Steps to get the theoretical $a \sim S$ relation as a function of film modulus and η

6. Assume the same initial values of M_f and η as in step 1.
7. Calculate the effective thickness t_{eff} for a given point on the indentation loading curve using Eq. 16.
8. Calculate the elastic $S \sim a$ relation directly from Yu's solution by using Eqs. (9)~(14).
9. Calculate the theoretical contact area a^* where the contact stiffness equals S_r (as from step 4), from the elastic $S \sim a$ relation.
10. Repeat step 6 through 9 for every point of the indentation loading curve to obtain the theoretical $\left[S_r, a^*\right]$ relation for the values of M_f and η assumed in step 1.

III. Steps to extract unknown film modulus

10. Compute the χ^2, sum of residues squared, using the following formula:

$$\chi^2 = \sum\left(a_{\exp} - a^*\right)^2$$

11. Find the values of M_{f} and η that minimize χ^2 using a standard optimization

algorithm.